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THERMO-DIFFUSION IN THE TUNGSTEN-BERYLLIUM SYSTEM

by

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1. Introduction

Of all the metals in nature tungsten possesses the highest melting temperature (3410°C). The elastic modulus of tungsten ($40,600\text{ kg/mm}^2$) exceeds that of such important metals for technology as tantalum, molybdenum, and niobium. The ability of tungsten to maintain satisfactory mechanical properties at elevated temperatures above 2000°C makes it attractive as a highly heat resistant material.

However, in a number of cases it has to be taken into consideration that tungsten begins to oxidize rapidly if heated to $400\text{--}500^{\circ}\text{C}$ in air. With further rise in temperature the oxidation of tungsten begins to become more intense; the trioxide of tungsten which forms on the surface of the metal at temperatures in excess of 800°C has a high vapor pressure, and therefore its evaporation begins.¹

We have conducted an investigation on surface alloying of tungsten by various elements separately and in combinations. It should be noted that only few works have been published on surface alloying of tungsten.²⁻⁵ The method of thermodiffusional surface alloying not only permits information on surface hardening of the metal, but also on the phase composition of systems, on diffusion processes, energy characteristics of the phases, and other information.

The present work presents data on the W-Be system. Beryllium is interesting because it possesses such properties as low specific weight for comparatively high durability, good stability against corrosion, and possesses relatively high temperature of melting (1315°C).⁶

The system W-Be has been studied very little. Reference 7 indicates the existence of a WBe_2 phase, having a hexagonal structure with lattice parameters $a = 4.437$, $c = 7.274\text{ kX}$, and another phase of composition MoBe_{12} . This phase has a volumetrically centered tetragonal structure with lattice parameter $a = 10.14$, $c = 4.23\text{ \AA}$. Reference 8 notes the existence of phase WBe_{20} , having a marginally centered cubic grid with a lattice parameter $a = 11.64\text{ \AA}$.

2. Surface Alloying

In the work on thermodiffusion of surface alloying, tungsten rods were used. They had a purity of 99.95 percent, were pressed, baked, and then hammered to a diameter of 10 mm. The microhardness of the rods was 450 kg/mm^2 . The thermodiffusional saturation of tungsten samples was conducted with beryllium of 99.8 percent purity, at temperatures of $900\text{--}1200^{\circ}\text{C}$ with various soaking periods.

Figure 1 shows the kinetics of the phases growth in the diffusion zone at constant temperature (1100°C) and variable duration of the process. From the graph it is evident that the diffusion proceeds according to a parabolic law. An identical dependence is manifested as well in a case of interaction of beryllium with molybdenum and niobium.^{9,10}

Dissimilarity in the thicknesses of various phases in the diffusion zone depends on several factors, and first of all on the rate of diffusion in the corresponding phase.

Utilizing the obtained data, it is possible to compute approximately temperature dependences of coefficients of diffusion in each phase:

for phase WBe_{24}

$$D = 1.175 \cdot 10^{-3} \exp (-33,800/RT) \quad (1)$$

for phase WBe_{12}

$$D = 2.36 \exp (-66,950/RT) \quad (2)$$

for phase WBe_2

$$D = 1.10 \cdot 10^{-4} \exp (-33,115/RT) . \quad (3)$$

In the process of saturation of beryllium, on the surface of the tungsten sample there forms a diffusion zone of complex composition and structure. The microstructure of diffusion layers, obtained at a saturation temperature of 1110°C in the course of four hours, is presented in Figure 2. An analogous structure is obtained as well with saturation temperatures of 900-1200°C.

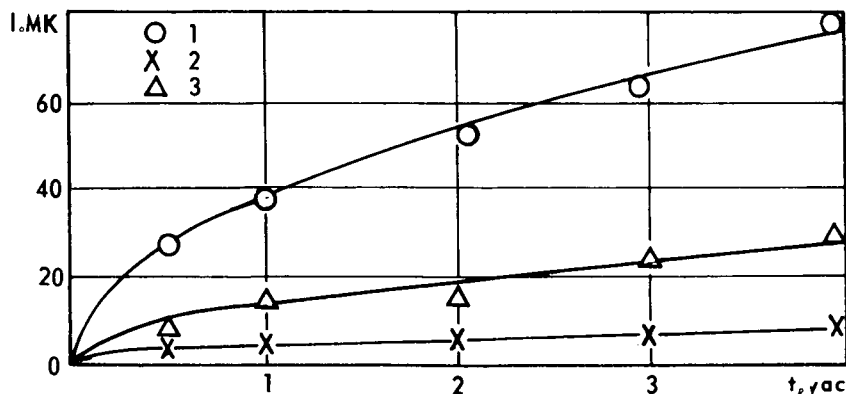


Figure 1. The Kinetics of Phase Growth in the Diffusion Zone at a Saturation Temperature of 1100°C:

1) First Layer, 2) Second Layer, and 3) Third Layer

Diffusion proceeds with a front with a simultaneous formation of several layers. The internal layer, directly adhering to the metal (tungsten), has clearly defined boundaries with jagged edges, testifying to the now uniform diffusion process in microsections.

A Roentgen-spectral analysis* has shown that the chemical composition of the internal layer corresponds to phase WBe_2 (Figure 3); X-ray investigations have shown that this layer has a crystalline structure type C14, hexagonal (spatial group $\text{P6}_3/\text{m}\mu$) with lattice parameters: $a = 4.446$, $b = 7.347$ kX. The structure of the internal diffusion layer and the lattice parameter determined by us agree with the data on phase WBe_2 , obtained through investigation of alloys in reference 7. The microhardness of the layer is very high: 2188 kg/mm^2 .

The second layer, adhering to phase WBe_2 from the outside is colored grey by the reagent, has somewhat uneven delineation of boundaries and gives evidence to the unevenness of diffusion in microvolumes. In the spectrogram curve (Figure 3) this phase is shown as a compound, corresponding to the chemical composition of formula WBe_{12} .

X-ray analysis shows that this layer represents a WBe_{12} compound having a body-centered tetragonal lattice with periods $a = 7.29$ and $c = 4.28$ kX. The structure type of C11a, C11c is spatial group $14 \text{ m}\mu^{-1}$.

Phase WBe_{12} which was discovered by us, corresponds to formula MoBe_{12} in chemical composition. However, the lattice parameters differ significantly from those indicated in reference 7. A check invariably verified our data. The deviations were insignificant, which is due to the small region of homogeneity of the WBe_{12} phase.

Comparison with the analogous beryllide of niobium NbBe_{12} , which also has volume-centered tetragonal lattice with lattice parameters of $a = 7.376$ and $c = 4.280$ kX¹⁰ i. e., close to the ones found by us for WBe_{12} , indicates the correctness of the WBe_{12} lattice parameter values that were obtained. The atomic radii of tungsten (1.40) and molybdenum (1.39) are close; taking into consideration the analogy in chemical properties of these elements, similar lattice parameters in analogous compounds are expected.

Thus, it may be considered credible that a WBe_{12} compound with volume-centered tetragonal structure exists and that it has the lattice parameters discovered by us. The microhardness of the WBe_{12} phase (1145 kg/mm^2) is significantly lower than the WBe_2 phase hardness.

*Analysis was conducted with participation of E. Marchukova.

The external layer, bright, weakly etched by the reagent, has a relatively great thickness. X-ray analysis of the layer indicates a chemical composition, corresponding to the WBe_{24} compound (Figure 3).

An X-ray pattern of the layer is indexed according to the tetragonal system. The computation gave the lattice parameters values of $a = 7.849$ and $c = 12.730$ kX.

In the diffusion zone of system W-Be, phase WBe_{20} with a cubic structure ($a = 11.64 \text{ \AA}$) indicated in reference 8, is not discovered. The phase found by us is formed in the diffusion zone in the form of a large layer. It has a far greater homogeneity area in comparison with other beryllides of tungsten and it would be admissible to consider the possibility of its inclusion in the WBe_{20} composition, but based on data contained in reference 8, the WBe_{20} compound has an entirely different crystalline structure than WBe_{24} . Consequently the problem of the existence of phase WBe_{20} remains unclear.

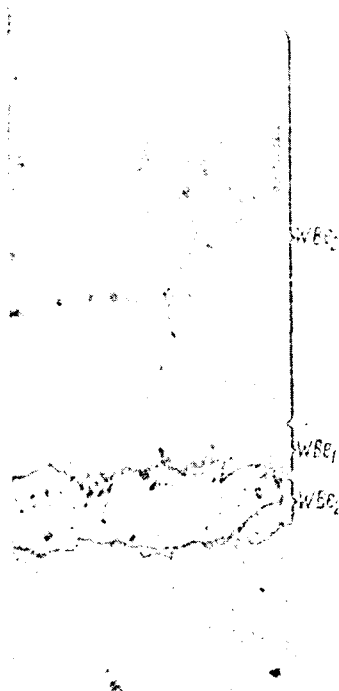


Figure 2. Diffusion Layer Microstructure on the Surface of a Tungsten Sample with Saturated Beryllium: 1100°C , Time 4 Hours (X 450)

The hardness of the WBe_{24} phase is equal to 1080 kg/mm^2 . It is less than the hardness of the WBe_2 and WBe_{12} compounds, but considerably greater than tungsten hardness.

The WBe_{24} phase layer consists of large columnar grains oriented in the direction of the diffusion flow. In places in the internal part of the layer, inclusions were noted that have an identical color with the adhering second layer. In color and X-ray analysis these inclusions may refer to the WBe_{12} phase. Their formation in the WBe_{24} external layer may be explained in two ways; first by the nonuniformity of beryllium diffusion in tungsten caused by the presence of dissolved admixtures and submicroscopic inclusions; second, by variation of the beryllium solubility boundary in the WBe_{24} phase in dependence on temperature. It is not difficult to conceive the solubility boundary variation in the direction of the beryllium concentration increase, as it is schematically shown in Figure 4.

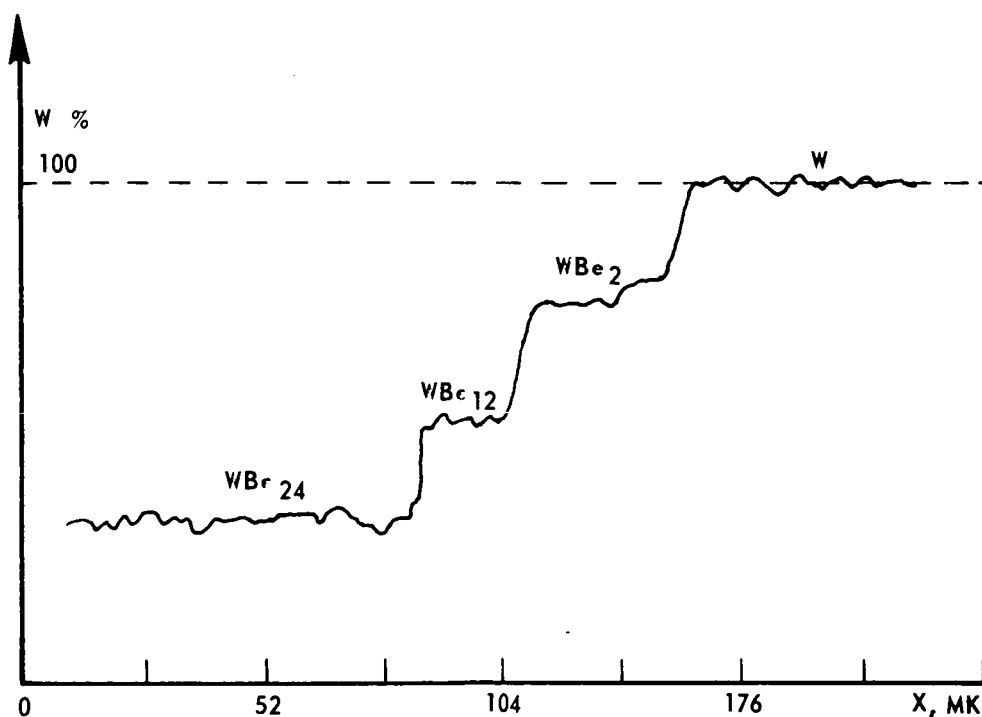


Figure 3. Tungsten Dispersion Curve Along Diffusion Zone Depth.

In this case, at diffusion temperature, the beryllium concentration in the diffusion layer may be obtained from a (Figure 4) on the internal boundary to c on the surface. On cooling after diffusional saturation the beryllium concentration in the WBe_{24} phase will change according to curve a'b. In the external diffusion layer, what remains single phase is only that part of the layer which has a concentration of beryllium within limits bc. The internal part of the layer, having

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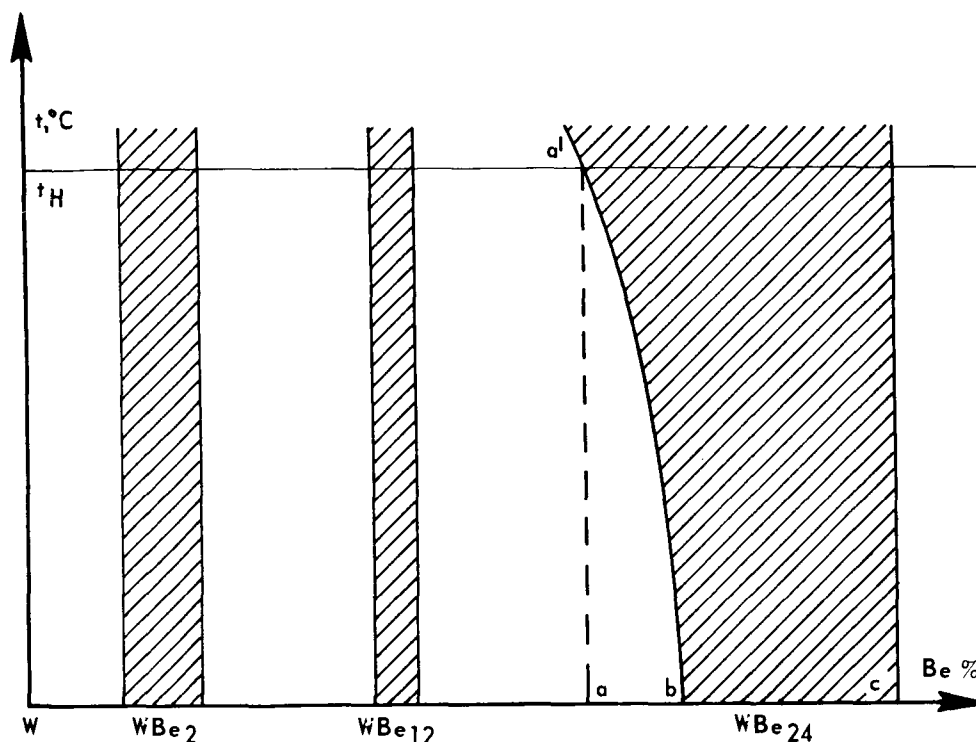


Figure 4. Schematic Representation of Part of a Diagram of the State of the W-Be System

the beryllium concentration within a-b limits becomes heterogeneous. Its base will be WBe_{24} phase, and in it, as in the matrix, will be located the precipitations of the second phase, WBe_{12} . Probably, such a figure was observed on the internal side of the diffusion layer.

The kinetics of tungsten oxidation were studied by many authors. As a result, a lot of experimental and theoretical material has been collected.¹¹⁻¹⁵ However, there is still little material on the study of oxidized tungsten saturated by various elements.

We have conducted the investigation of oxidation of tungsten saturated by beryllium. The kinetics of oxidation was studied in an atmosphere of air at

900-1200°C by the method of continuous weighing with an accuracy of ± 0.0005 g. The duration of oxidation was 10 hours, with measurements every 15 minutes for the first two hours of oxidation, and every half hour after that.

On the surface of the samples at all investigated temperatures (1000-1200°C) there forms a thin oxide film (10-15 microns) white-grey in color, closely bonded with basic metal. In interpreting the electrograph of the oxide film, data on the magnitude of the free energy of the beryllium and tungsten oxide formation (BeO -140 Kcal, $1/3 \text{ WO}_3$ -60.6 Kcal) were taken into consideration. From these data it is apparent that beryllium has a greater affinity for oxygen than for tungsten (almost 2.5 times).

As has been shown by electrographic analysis, the oxide layer consists of BeO oxide with a hexagonal lattice and a period $a = 2.694$, $c = 4.392$ kX. The structure is type B4 spatially homogeneous group $\text{P6}_3\mu$.

3. Conclusion

Beryllium and tungsten diffusion at 1000-1200°C proceeds with a front with a simultaneous formation of a number of intermetallic phases of various thickness. The phases are separated between themselves while their boundaries correspond to steps in the concentration curve.

The temperature dependences of the diffusion coefficients have been obtained for each phase.

By means of X-ray and X-ray spectral analysis, the structures and the phase composition of the diffusion layer have been studied. Beryllides of tungsten of the following stoichiometric composition have been established: WBe_{24} , WBe_{12} , and WBe_2 . The crystal structures of these phases have been established.

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